



## PATENT SPECIFICATION

DRAWINGS ATTACHED

1,199,190

Date of Application (No. 51799/67) and filing Complete Specification: 14 Nov., 1967.

Two Applications made in United States of America (Nos. 593,658 and 594,024) on 14 Nov., 1966.

Two Applications made in United States of America (No. 660,411 and 660,583) on 15 Aug., 1967.

Application made in United States of America (No. 693,757) on 6 Oct., 1967.

Complete Specification Published: 15 July, 1970.

Index at acceptance:—D1 R1

International Classification:—D 04 h 1/42, 1/72

## COMPLETE SPECIFICATION

## Absorbent Non-Woven Fabrics

We, E. I. DU PONT DE NEMOURS AND COMPANY, a Corporation organised and existing under the laws of the state of Delaware, United States of America of, Wilmington 98, Delaware, United States of America, do hereby declare the invention for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to an absorbent non-woven fabric and to a process for preparing the fabric.

Nonwoven fabrics deriving their strength from fibre entanglement are disclosed in Belgian Specification No. 673,199. The absorbent fabric of this invention is an improvement over the fabrics disclosed in the latter Specification since it retains its strength in the presence of urine and body fluids, but is water-dispersible. A flushable fabric is thus provided.

Thus, the invention provides inexpensive, soft, absorbent, textile-like structures which are readily disposable in sewage systems, as by flushing down a household toilet drain. They are suitable for use as sanitary fabrics, diapers, bandages and panties, where absorbency for body fluids, ease of disposal after a single use, pleasing appearance and soft tactile hand are required. They have adequate strength and surface stability for such uses.

The fabric of the invention is a fabric of textile fibres secured in place by random fibre entanglement to provide dry strength and integrity in the absence of resinous or fusion bonds, the fabric comprising from 10 to 100% water-sensitive fibres (as defined herein) of less than three inches in length, the balance of the fibres consisting of non-water-sensitive fibres of less than 0.5 inch in length,

the fabric having a  $C_{h \text{ dry}}$  value (as defined herein) of at least 0.2 and a  $C_{h \text{ wet}}$  value (as defined herein) of less than 0.3. In the fabrics, the ratio

$$\frac{C_{h \text{ dry}}}{C_{h \text{ wet}}} \geq 1.3$$

Preferably the fabric has a weight of 0.3—5 oz/yd<sup>2</sup> and most preferably has an average wet strip tensile strength of less than 0.1 lb/in. It is particularly preferred that the fabrics have an elongation at break, in two directions at right angles to each other, of at least 20%, the  $C_{h \text{ wet}}$  is less than 0.15 and the fabric contains at least 40% water-sensitive fibres. Most preferably, the fabric has an average tensile strength in synthetic urine (as hereinafter defined) of at least 0.05 lb/in., a  $C_{h \text{ dry}}$  of at least 0.3,  $C_{h \text{ wet}}$  not more than 0.15 and a ratio of  $C_{h \text{ dry}}$  to  $C_{h \text{ wet}}$  of at least 2. It is also preferred that the water-sensitive fibres have a denier per filament of 0.5 to 15 and a length 0.25 to 2 inches. The preferred water-sensitive fibres are those of cellulosic esters and ethers containing —OA groups where A is the substituent, wherein the corresponding AOH compound is soluble in water at 20°C to an extent of at least 3%. Two highly preferred fabrics consist either (a) substantially of water-sensitive fibres or (b) are layered, the surface layer containing at least 80% non-water-sensitive fibres and the centre layer containing at least 70% water-sensitive fibres, the overall structure comprising 30—90% water-sensitive cellulosic fibres.

The invention also provides a process for preparing the fabrics, the process comprising supporting a non-woven web comprising at least 10% of fibres which are water-sensitive

[Price 5s. 0d. (25p)]

or are capable of being rendered water-sensitive, and are less than 3 inches in length, the remainder of the fibres being non-water-sensitive and not more than 0.5 inch in length, on an apertured member, directing fine essentially columnar streams of a liquid essentially inert to the fibres at an energy flux of at least 23,000 ft.-poundals/in.<sup>2</sup>-sec. at the web so as to impinge thereon, and moving the web relative to the streams, with the proviso that when the fibres in the web are water-sensitive before directing the stream thereat, the stream comprises a de-swelling liquid for water-sensitive fibres and has a Y value (as defined herein) of  $3 \times 10^6$  to  $25 \times 10^6$  ft./min.; and then if necessary rendering water-sensitive the fibres capable of being so rendered.

The term "Y value", as used herein, is the weight of liquid used in lbs./min.  $\times$  the liquid velocity ft./min. divided by the weight of the fibrous material treated in lbs./min.

Preferably, the fibres are rendered water-sensitive by applying to the fabric a 17—30% solution of an amic acid (as hereinafter defined) in an amount 100 to 300% by weight of dry fibres, and heating the fabric at 165—210°C, or by applying to the fabric a mixture of phosphoric acid and urea (in a ratio of 1:3 to 1:2) in an amount of 60—160% by weight of dry fibres, and heating the fabric at 125—210°C.

The coherence value ( $C_h$ ) is the degree to which the fabric, when wet ( $C_{h \text{ wet}}$ ) or dry ( $C_{h \text{ dry}}$ ), comes apart by fibre-to-fibre slippage as opposed to fibre breakage whenever a long strip of the fabric is pulled apart and is thus a measure of interfibre friction from which the fabric derives its strength. In this specification, the coherence values are measured in the absence of resinous or fusion bonds.

The term "nonwoven fabric" as used herein means a structure having a drape as measured by a bending length (see page 28, lines 24—28) of less than 3.0 cm. in two directions at right angles to each other and elongation-at-break of at least 9% in at least one direction.

Water-sensitive fibres are those with a wet coefficient of sliding friction of less than 0.90 (preferably less than 0.70) as measure on a nonwoven fabric consisting essentially of the salt free fibres being tested, which fibres must also be insoluble in synthetic urine.

The fibres may be homogeneous, i.e. have a substantially uniform chemical composition throughout their cross-section, or they may be heterogeneous, thereby having an adherent coating or skin of a water-sensitive composition.

Synthetic urine is a salt solution containing 10 g. sodium chloride, 24 g. urea, 6.0 g. magnesium sulphate and 0.7 g. calcium acetate monohydrate per litre of solution in distilled water.

When the fabric has a  $C_{h \text{ wet}}$  of less than about 0.30, the entangled fibres break up into

smaller pieces in water after dipping by the relatively mild forces of the turbulent flow in a toilet bowl.

The preferred fabrics have  $C_h$  (wet) values of less than about 0.15. These fabrics are flushable, in the form of relatively large fabrics (such as 13 in.  $\times$  17 in.) with reduced turbulence of water, without the need of pre-dipping before flushing.

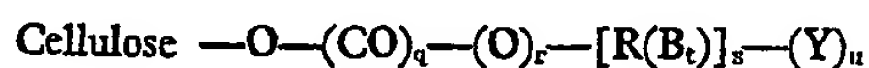
Some water-sensitive fibres useful in this invention are well known.

An easy and well-known route to such products is by modifying cellulose. Suitable chemical reactions involving derivatives of the hydroxyl group are discussed in Ott and Spurlin, "Cellulose" Vol. II, pages 673 to 1026 (Interscience Publishers, Inc., New York, 1954) and water-soluble derivatives given therein. Oxycellulose can also be used. Further products are cited in Reinhardt *et al*, Textile Research Journal, 1957, 27, 59—65. The chemical modification of cellulose and other polymers is discussed at length in Fettes "Chemical Reactions of Polymers" (Interscience Publishers, New York, 1964).

The reactions, such as esterification and etherification, used to convert cellulose to a water-sensitive derivative can be applied to non-cellulosic polymers that have reactive hydroxyl groups along their chains.

Water-sensitive fibres are also readily obtained by hydrolysing polymers in fibre form such as polyvinyl acetate (to give polyvinyl alcohol) or copolymers of vinyl acetate with monomers such as ethylene, vinyl chloride and methyl methacrylate.

Most preferably the water-sensitive fibres are chemically modified cellulosic fibres having a degree of substitution (hereinafter referred to as "D.S.") at the surface of 0.1 to 1.5 and represented by the general formula



wherein R is basically a hydrocarbon group free from aliphatic unsaturation which may contain non-interfering hydrophobic substituents, ether oxygen linkages and ester oxygen linkages in the chain; B is a hydrophilic substituent; Y is hydrogen atom, an acid group having ionizable hydrogen attached to an oxygen atom or a monovalent cation salt of the acid (subject to the provisos that when Y is a hydrogen atom and t is zero, R has less than 5 carbon atoms; when Y is hydrogen and t is one, R has less than 8 carbon atoms; when Y is a salt and t is zero, R has less than 19 carbon atoms but when Y is an acid group and t is zero, R then has less than 10 carbon atoms); q, r, and s are zero or one but when r is one, q and s are one; t is zero or any integer up to the number of replaceable hydrogen atoms in R, and u is 1 or 2.

Suitable hydrophilic groups for the cellulose ether or ester derivatives include sulphonyl,

5 sulphonamide, phosphoramidate, phosphonamide, hydroxyl, carbamyl, amino, lower alkyl substituted amino (low molecular weight groups less than about 80). These groups are those which increase the water solubility of a given hydrocarbon when replacing a hydrogen atom.

10 Suitable acid groups for the cellulose ethers and esters include those groups having oxygen connected directly to carbon, sulphur or phosphorus (with molecular weights less than about 100). These acidic groups have an ionization constant for the first hydrogen atom of at least  $1 \times 10^{-5}$ .

15 The hydrophobic or non-interfering substituents are those groups such as halogen, nitrile and nitro groups, that have little effect on the water-sensitivity of a cellulose compound but which may be present to a limited extent due to the synthesis used.

25 Suitable cellulose ethers include lower alkyl ethers such as methyl-, ethyl-, propyl-ethers. Substituted ethers such as cyanoethyl-, cyanomethyl-, hydroxyethyl-, acrylamido-, aminoethyl-, dimethylaminoethyl-, ethylaminoethyl-, dinitromethyl-, poly(ethylene oxide)-ethers, may also be used. Preferred cellulose ethers containing acid groups include carboxymethyl-, carboxyethyl-, sulphonyethyl-, sulphonybutyl-, sulphatoethyl-, phosphopropyl-, phosphatoethyl-, malonic-, carboxybenzyl-, sulphonybenzyl-ethers.

40 Suitable cellulose esters include the lower alkyl esters such as the formates, acetates and propionates, and substituted esters such as hydroxyacetate, hydroxybutyrate,  $\alpha$ -hydroxy dimethyl propionate, ethyl carbonate, to mention a few.

45 Preferred cellulose esters containing an acid group include cellulose phosphate, sulphate, sulphite, phosphite, borate, hemioxalate, hemimalonate, hemisuccinate, hemiglutarate and hemiphthalate.

The above basic esters and ether-forming groups may have a wide variety of other substituents as desired. 50

In general the highest water-sensitivity for a given D.S. for a given compound is observed with fibres made by extruding a solution of the desired derivative. Thus extruded fibres of cyanoethylcellulose of D.S. 0.15 to 0.80, hydroxyethylcellulose of D.S. 0.1 to 0.9, sodium carboxymethylcellulose of D.S. 0.1 to 0.3 and sodium carboxyethylcellulose of D.S. 0.1 to 0.3 are suitable. Preparation of these fibres is known in the art. The cyanoethylated fibres are discussed in French Specification No. 1,346,262. 55

Suitable fibres made by chemical modification of cellulose fibres include sodium carboxymethylcellulose of D.S. 0.2 to 0.4, sodium cellulose sulphate of D.S. 0.2 to 0.4, monosodium cellulose phosphate of D.S. 0.2 to 0.4 and sodium cellulose hemisuccinate of D.S. 0.2 to 0.4. In general, higher D.S. values are required for this type of fibre to obtain a given degree of water sensitivity than with the extruded fibres. The D.S. ranges for the above sodium salts are for completely neutralized derivatives. Partially neutralized or partially cross-linked (e.g., by polyvalent cations) derivatives of higher D.S. values can also provide the desired water sensitivity. 60 65 70 75

One means of deciding whether a cellulose ester or ether containing —OA groups is suitable is to prepare the compound having the formula AOH and test its solubility. If the AOH has a solubility in water at 20°C. of at least 3%, then the cellulosic ether or ester is suitable for use as a water-sensitive fibre in this invention. 80 85

The group A can be an organic or inorganic group or a combination of organic and inorganic groups as shown below: 90

Cellulose ethers	A—OH	Solubility
n-butyl —	n-butyl alcohol	7.9
cyanoethyl —	cyanoethyl alcohol	∞
aminobutyl —	2-amino butanol-1	∞
carboxypropyl —	α-OH butyric acid	>20
Cellulose esters		
cellulose propionate	propionic acid	∞
cellulose hemisuccinate	succinic acid	6.8
cellulose phosphate	phosphoric acid	∞
cellulose sulphate	sulphuric acid	∞
potassium cellulose phthalate	potassium acid phthalate	10

Preferably, the group "A" contains or consists entirely of an acidic group, as a free acid or partially or completely in the form of a salt of an alkali metal or ammonium ion. Such groups include —COOH, —PO(OH)<sub>2</sub>, —OP(OH)<sub>2</sub>, —OSO<sub>2</sub>OH, —SO<sub>2</sub>OH, —OB(OH)<sub>2</sub> attached to a carbon atom, or groups such as —PO(OH)<sub>2</sub>, —P(OH)<sub>2</sub>, and —SO<sub>2</sub>OH which may compose all of group A and are connected to the cellulose through oxygen.

Another route to the water-sensitive fibres is to hydrolyse polymers having pendant side groups of esters, amides and nitriles to carboxyl groups or amides.

Polymers and copolymers from acrylonitrile, alkyl acrylates, alkyl methacrylates, acrylamides and vinyl phosphonates are suitable for this purpose. The same type of polymer can also be made by copolymerizing acidic monomers such as acrylic acid, methacrylic acid maleic anhydride, styrene sulphonic acid, and vinyl sulphonic acid with another monomer such as acrylonitrile, styrene, vinyl chloride or vinylidene chloride. The combinations of monomers and the appropriate polymerization techniques are well known.

Another route is to start with known water-soluble fibres and cross-link them by ionic bonds (such as calcium ion on an acid polymer), or by covalent bonds with difunctional reactants (diamines, glycols, etc.) or by the application of high energy ionizing radiation (e.g. by a 2 MEV Vandegraff electron accelerator). The water solubility of these fibres can also be reduced by increasing the molecular orientation e.g. by further drawing. An example of such a fibre is a polyvinyl alcohol fibre.

Another route to the water-sensitive fibres is the bonding of acidic compounds to fibres by irradiation as taught in U.S. Patent Specification No. 2,999,056.

Many condensation polymers are known to be water-soluble and these can be utilized in the present invention if the other criteria are met.

These polymers can be cross-linked to reduce their water-solubility and still retain adequate water-sensitivity, or the polymers can be made as copolymers with condensation monomers such as hexamethylene diamine and adipic acid to give a water-sensitive polymer directly.

Typical nonwater-sensitive fibres are the usual textile fibres of commerce (e.g. rayon, cotton, nylon, acrylic or polyester). Fibres of 1 to 5 denier are most satisfactory but smaller fibres such as paper-making fibres and cotton linters can be used. Preferably the fibres are biodegradable since the products are to be dispersed in sewage systems. Cellulosic fibres such as cotton or viscose rayon are especially useful.

In the accompanying drawings,

Figure 1 is a schematic view of apparatus suitable for preparing the fabrics,

Figure 2 is a schematic isometric view of an apparatus for high-speed, continuous production of the fabrics,

Figure 3 is a photomicrograph at 10X magnification of a representative portion of the fabric prepared according to Example I, hereinafter, showing the top or upstream face of the fabric by direct illumination,

Figure 4 is a photomicrograph at 10X magnification corresponding to Figure 3, but



showing the bottom face of the fabric which is next to the screen support during production,

5 Figure 5 is a photomicrograph at 10X magnification of a representative portion of one type of patterned entangled fabric showing the top face of the fabric by direct illumination, and

10 Figure 6 is a photomicrograph at 10X magnification corresponding to Figure 5, but showing the bottom face of the fabric.

15 Figures 3 to 6 illustrate fabrics of the invention wherein fibres are secured in place by random fibre entanglement in a repeating pattern of ordered fibre groups arranged in parallel rows interconnected by fibres extending between adjacent rows to form a unitary structure. A wide variety of such patterns can be prepared as disclosed in Belgian Patent Specification No. 673,199. The fabric of 20 Figures 3 and 4 is prepared on a plain weave, 24 x 24 mesh/inch woven wire screen. The resulting fabric has ordered fibre groups arranged in regular parallel rows extending in two principal fabric directions and interconnected in a network by fibres randomly entangled with each other to defined an ordered 25 geometric pattern of apertures. Fibre groups which form ridge-like protrusions on the face of the fabric are seen in Figure 4. In this embodiment, the ordered fibre groups follow paths located between adjacent parallel wires of the screen.

30 The fabric of Figures 5 and 6 is prepared on a grid of 40 mil diameter parallel rods spaced 12 to the inch. The repeating pattern of the fabric is characterized by groups of entangled fibres forming a regular pattern of ridge-like protrusions separated by grooves lying along parallel straight lines, the ridge-forming groups being interconnected by arrays of generally parallel fibres bridging under the ridge-separating grooves and entangled in adjacent groups.

35 A somewhat similar fabric can be prepared on oblong screens having 5 to 12 heavy wires per inch in one direction and 3 to 5 times as many finer wires per inch in the other screen direction.

40 Useful fabrics which have no repeating pattern of ordered fibre groups or apertures can also be made; they can resemble felts, for example. Such fabrics can be made by using fine mesh screens, for example with 80 or more wires per inch as a support for the fibres during entanglement.

45 The fabrics of the invention can be produced by treating webs containing water-sensitive fibres with essentially columnar (i.e. having a total divergence of not greater than 5 degrees) streams of a liquid exerting an energy flux of at least 23,000 foot-pounds per square inch-seconds at the web.

50 The energy flux (EF) of a stream can be calculated by means of the formula:

$$EF = 77 PG/A \text{ ft-pounds/in}^2 \text{ sec.} \quad 65$$

where P is the pressure at which the liquid is jetted in pounds per square inch gauge (psi); G is the average volume flow for one jet stream in cubic feet per minute and A is the cross-sectional area of the streams in square inches at a location just prior to impact with the fibrous webs. This area can be determined from photographs of the stream with the webs removed, or by means of micrometer probes. Suitable streams can be obtained by propelling a suitable liquid at high pressures through small-diameter orifices under conditions such that the emerging streams remain essentially columnar at least until they strike the fibrous material. The intensity of the streams is sufficient to entangle the fibres firmly in place.

70 A critical control of the extent of treatment is necessary to produce the fabric of this invention, taking into account the rate of application of energy by the streams, the rate at which the fibrous layer is traversed and the weight of the layer; the required extent of processing is defined by the Y value (see above). 75 When the fibrous material is treated a plurality of times, the Y value is the sum of the values calculated for each individual treatment. The total treatment conditions should be adjusted to provide a Y value of  $3 \times 10^3$  to  $24 \times 10^6$  ft./min. whenever low-swelling fibres are present in the fabric. The weight of liquid used and the weight of fibrous material treated are measured for a convenient time interval. The liquid velocity is then calculated 80 from the lb. liquid/min. by dividing by the density of the liquid in lbs./ft.<sup>3</sup> and by the total orifice area in ft.<sup>2</sup>.

85 A deswelling liquid is used in the streams to avoid swelling the water-sensitive fibres during treatment which would result in damage. A variety of aqueous salt solutions have been found suitable including sodium sulphate (17—20%), ammonium sulphate (20—30%), and sodium citrate (30%). Non-aqueous liquids can also be used.

90 Products of this invention can also be prepared from a web of non-water-sensitive fibre, entangling the fibres and then chemically modifying some or all of the fibres in the fabric to a water-sensitive form. For example, the cellulose can be reacted with chloracetic acid and alkali to produce carboxymethyl cellulose, with alkali and ethylene oxide to produce hydroxyethyl cellulose, with alkali and acrylamide or acrylonitrile to produce carboxyethyl cellulose, with phosphoric acid and urea to produce cellulose acid phosphate, with succinic acid to produce cellulose hemisuccinate, and with urea and sulphuric acid (or sulphamic acid) to produce cellulose sulphate; Such reactions work well with a regenerated cellulose such as rayon; with cotton 125

it may be necessary to degrade the cotton, mercerize it or use stronger reaction conditions.

Other reactions which are suitable for preparing water-sensitive fibres from non-cellulosic fibres are for example the hydrolysis of polyvinyl acetate fibres to polyvinyl alcohol the hydrolysis of cross-linked polyvinyl alcohol fibres, hydrolysis of acrylic fibres, and the use of the above-mentioned reactions for rayon on noncellulosic fibres having hydroxyl groups such as copolymers containing vinyl alcohol units.

The modified fabric is usually treated to remove excess reactants and by-products. The water sensitivity of the modified fibres must be considered when selecting the exact treatment. If the water-sensitivity of the modified fibres is due to a non-ionizing chemical group such as cyanoethyl or hydroxyl, the treatment can be done with a organic liquid such as alcohol or acetone but preferably is done with a fairly concentrated solution of a neutral salt. If the water-sensitivity of the modified fibres is due to an ionizing chemical group such as a weak acid, a different purification treatment can be used. The fibre is inert towards water when in the acid form such as (acid)carboxymethyl cellulose. Some reactions using acids give the fibres in the free acid form and such fibres can be washed in water. Other reactions using caustic for example yield the salt of the acid and the fibres can be treated with a strong acid such as sulphuric acid and then washed with water. The purified acid forms of the fibres are then converted to the salt form by neutralizing with a mild alkali, preferably a buffered system in the presence of a fairly concentrated salt solution or in a non-aqueous system. The modified fabric can be washed with a buffered solution of the proper pH and concentrated salt solution to yield the desired salt form.

Preferred processes are the following: (a) Treatment of nonwoven fabrics of regenerated cellulose with an aqueous solution containing 5 to 30% (preferably 8 to 20%) of sodium chloracetate and 2 to 10% (preferably 2 to 5%) of an alkali metal hydroxide to leave a pickup (see below) of from about 2 to 4 and subsequently drying the fabric at 100°C. to 180°C. for 0.1 to 5 minutes. This usually gives a degree of substitution of 0.2 to 0.4. The modified fabric must be treated to remove excess caustic and leave the etherified fibres in the form of sodium carboxymethyl cellulose. (b) Applying an amic acid (i.e. an organic acid having a carboxyl group atom end and a —CONH<sub>2</sub> group at the draw) to the nonwoven fabric of regenerated cellulose and heating to cause the reaction. The amic acid is conveniently applied as an aqueous solution in concentrations of 17 to 30% to give a pickup (i.e. weight of liquid taken up per unit weight of original dry fabric), of 1 to 3. This pickup

range can be increased depending upon the particular combination of cellulose fibre, amic acid, presence of catalyst and temperature of reaction. A catalyst or reaction promotor, such as sulphamic acid, p-toluenesulphonic acid, zinc chloride and magnesium chloride, may be used in amounts of 0.1 to 10% of the weight of the amic acid. Subsequent heating at 165° to 210° for 1 to 3 minutes have been found convenient. The resultant fibres are relatively inert to water and can be washed free from excess reagents and by-products with water without damage.

(c) Treatment of a nonwoven fabric of regenerated cellulose fibres with 0.6 to 1.6 times the weight of dry fibres with phosphoric acid and urea, applied as an aqueous solution containing 15 to 90% water, the weight ratio of acid, calculated as phosphoric acid, to urea being 1:3 to 1:2. The impregnated fabric is then heated at 125°C. to 210°C. until the cellulosic fibres have a degree of substitution of 0.2 to 0.4. The modified fabric can be purified by washing with water and then converted to a water-sensitive form by at least partially neutralizing with a slightly alkaline buffered solution containing a deswelling salt such as sodium sulphate.

The treatment energy expended during one passage of a fibrous web under a plurality of like jet streams in the preparation of a given nonwoven fabric, in horsepower hours per pound of fabric, may be calculated from the formula:

$$E_1 = 0.125 (XPG/sb) \text{ HP hr./lb.}, \quad 100$$

where:

- X = number of streams per linear inch of treatment width,
- P = pressure at which the liquid is jetted in pounds per square inch gauge (psi) 105
- G = average volumetric flow for one jet stream in cubic feet per minute,
- s = speed of passage of the web under the streams in feet per minute, and 110
- b = the weight of fabric produced in ounces per square yard.

The total amount of energy expended in treating the web is the sum of the values calculated as above for each pass under such streams, if there is more than one pass. The value of G for use in the above formula can be determined from flow rate measurements. 115

The treatment energy should preferably be at least 0.1 horsepower hr./lb. 120

The initial layer of fibrous material may be prepared as follows. It may be a web or batt of fibres assembled by known methods such as carding, random laydown, air deposition or by paper-making processes. The fibres may be disposed in random relationship with one another or in 125

any degree of alignment. A plurality of layers of the same or different composition or orientation can be used. When prepared by assembling dry fibres, the fibres can be up to about 3 inches in length in the final assembly. Preferably, the initial layer is formed on a papermaking machine, such as a Fourdrinier machine, from a slurry of water-sensitive fibres to 1 to 15 denier and 0.25 to 2.0 inch staple length. Conventional textile fibres of 1 to 5 denier and 0.125 to 0.5 inch staple length can also be included in the slurry. Slurries of staple fibres over 0.25 inch in length may be conveniently processed on a drum-type paper machine such as the "Rotoformer" manufactured by the Sandy Hill Corp., 29 Allen Street, Hudson Falls, New York. Conventional short wood pulp fibres may be substituted for part or all of the textile fibres in the slurry.

When a papermaking technique is used to form the sheet, the slurry consistency given by 0.1 to 0.40% fibres is suitable unless the fibres contain less than 30% of water-sensitive fibres. Suitable slurries containing a high proportion of nonwater-sensitive fibres, e.g., 90% of conventional textile fibres, are given by consistencies of 0.03 to 0.09% fibre. The slurry is fed to the headbox of the papermaking machine and is diluted with about an equal volume of water *en route* to the headbox. A double headbox supplied with different compositions of stock slurry can be used to prepare a laminated initial layer. From the headbox, the slurry is deposited on the wire of the papermaking machine to form a fibre layer.

The fibres drain as they are conveyed on the machine wire, the drainage being assisted by the usual suction boxes. The partially drained layer of fibres is sprayed with a 17 to 20% aqueous sodium sulphate solution, or other deswelling liquid, to cause deswelling of the highly water-absorbent fibres in the layer. For example, the layer may be sprayed with banks of nozzles at 2, 3 and 4.75 feet from the breast roll of the machine, using about 1.3 gallons per minute of the deswelling liquid. The deswollen layer passes from a vacuum couch roll to a pressing section where water and excess salt are removed. The layer may be dried on rolls heated to 120°C. and stored for subsequent treatment. Alternatively, the damp layer can be passed directly to the fluid treatment with high energy flux streams to form the products of this invention.

A relatively simple form of equipment for entangling the fibres in the fibrous layers with essentially columnar streams at the required high pressure is illustrated in Figure 1 and is fully described in Luxembourg Patent Specification No. 46,703. Liquid at a pressure of approximately 70 pounds per square inch (psi.) is supplied through valve 1 and pipe 2 to a high-pressure hydraulic pump 3. The

pump may be a double-acting, single-plunger pump operated by air from line 4 (source not shown) through pressure-regulating valve 5. Air is exhausted from the pump through line 6. Liquid at the desired pressure is discharged from the pump through line 7. A hydraulic accumulator 8 is connected to the high-pressure liquid line 7. The accumulator serves to even out pulsations and fluctuations in pressure from the pump 3. The accumulator is separated into two chambers 9 and 10 by a flexible diaphragm 11. Chamber 10 is filled with nitrogen at a pressure of one-third to two-thirds of the desired operating liquid pressure and chamber 9 is then filled with liquid from pump 3. Nitrogen is supplied through pipe 12 and valve 13 from a nitrogen bottle 14 equipped with regulating valve 15. Nitrogen pressure can be released from system through valve 16. Liquid at the desired pressure is delivered through valve 17 and pipe 18 to manifold 19 supplying orifices 20. Fine, essentially columnar streams of liquid 21 emerge from orifices 20 and impinge on the fibrous material 22 supported on apertured patterning member 23.

The streams are traversed over the fibrous material, by moving the patterning member 23 and/or the manifold 19, to entangle the fibres into a patterned structure at high energy flux throughout the area to be treated. In general, it is preferred that the initial fibrous layer be treated by moving patterning layer 23 under a number of fine, essentially columnar streams, spaced apart across the width of the material being treated. Rows or banks of such spaced-apart streams can be utilized for more rapid, continuous production of entangled fabrics. Such banks may be at right angles to the direction of travel of the web, or at other angles, and may be arranged to oscillate to provide more uniform treatment. Streams of progressively increasing energy flux may be impinged on the fibrous material during travel under the banks. The streams may be made to rotate or oscillate during production of the patterned structures, may be of steady or pulsating flow, and may be directed perpendicular to the plane of the layer of fibres, or at other angles, provided that they impinge on the fibres at a sufficiently high energy flux.

Apparatus suitable for use in the continuous treatment of fibrous layers to entangle the fibres into the fabrics of the present invention is shown in Figure 2. A fibrous layer 29 on apertured patterning member 30 is supplied continuously to moving carrier belt 31 of flexible foraminous material such as a screen. The carrier belt is supported on two or more rolls 32 and 33 provided with suitable driving means (not shown) for moving the belt forward continuously. Six banks of orifice manifolds are supported above the belt to impinge liquid streams 34 on the fibrous layer at successive positions during its travel



on the carrier belt. The fibrous layer passes first under orifice manifolds 35 and 36, which are adjustably mounted. Orifice manifolds 37, 38, 39 and 40 are adjustably mounted on frame 41. One end of the frame is supported for movement on a bearing 42, which is fixed in position. The opposite end of the frame is supported on oscillator means 43 for moving the frame back and forth across the fibrous layer to provide more uniform treatment.

High pressure liquid is supplied to the orifice manifolds through pipe 18, as in Figure 1. Each manifold is connected to pipe 18 through a separate line which includes flexible tubing 44, a needle valve 45 for adjusting the pressure, a pressure gauge 46, and a filter 47 to protect the valve from foreign particles. The conditions are readily adjusted to provide the desired treatment of different initial fibrous layers.

The pattern produced by the hydraulic entangling process is dependent upon the design of the apertured patterning member. The term "apertured member", as used herein, includes any screen, perforated or grooved plate on which the fibre assembly is supported during the hydraulic entangling and which by reason of its apertures and/or surface contours influences the movement of fibres into a pattern in response to the fluid streams. The apertured member may have a planar or nonplanar surface or a combination of the two types.

Plain weave screens of from 3 to 80 wires per inch having wire diameters of from 0.005 to 0.040 inch, and having 10% to 98% open area are suitable apertured members and in general afford the type of pattern shown in Figure 3.

Twill weave screens and Dutch twill of the above general construction may also be used.

An assembly of a grid of parallel rods contiguous with a second grid and so oriented that the rods of each grid are not parallel to the rods of the other grid can be used as an apertured member. Structures of the type shown in Figures 5 and 6 can be made using such supports.

The fabrics of this invention are capable of many modifications. They can be tinted, printed with designs, and made in a wide variety of patterns.

In addition to the use of simple nonwoven fabrics of this invention for sanitary purposes, various other combinations are possible. The nonwoven fabric may be used as a cover for a very high absorbency pad for special applications.

Papers made by deposition of fibres on a moving screen such as a Fourdrinier machine usually show different properties in the continuous direction (i.e., direction of travel on the paper machine) termed "machine direction" (MD) and in the direction transverse to travel termed "cross direction" (CD). These

terms are also applied in a like manner to nonwoven fabrics made by passing a long web of fibres under a hydraulic entangling assembly in one direction (MD). Products made from hand paper sheets (i.e. paper-like sheets that are made on a small scale laboratory apparatus) usually have very similar properties in any two directions at right angles and the direction is not designated.

The tensile strengths and elongations are measured on an Instron ("Instron" is a Registered Trade Mark) testing machine using a 2-inch length between the jaws and an elongation rate of 50% per minute. Results for Examples 1 to VI below are determined on 0.5 inch wide strips while the remainder are determined on 1.0 inch wide strips. In some cases, the breaking force determined in the coherence value test is used to calculate the tensile strength; such values are marked with "c". All tensile strengths are quoted in pounds/inch of width. All three methods give approximately the same results.

Samples are soaked for 5 minutes in distilled water at room temperature, then clamped in the tester and broken in air to determine wet tensile strength. These values are referred to as the wet strip-tensile strength.

The *coherence value* ( $C_h$ ) is a measure of the degree to which the fibres within the fabric break as opposed to slipping apart whenever a long strip of the fabric is pulled apart.

The test comprises the tensile strength of a strip (of length shown at page 26 lines 20—23 below) of the fabric and dividing this value by the tensile strength of the strip as determined at substantially zero length (Instron clamping jaws together). It is readily apparent that, during the zero length strip-tensile measurement, fibre slippage cannot occur and hence the maximum tensile strength for the strip is obtained. Therefore the maximum  $C_h$  value is 1 with lower  $C_h$  values indicating fibre slippage. A  $C_{h,wet}$  of less than about 0.3 is necessary to achieve the desired dispersibility of the fabrics of this invention, while a  $C_{h,dry}$  of at least 0.2 is required to yield adequate in-use strength.

Samples of salt-free fabrics are cut into 1.6 inch wide strips. The strips are then cut to about 2-inch and 1-inch long adjacent samples. The longer sample is broken using 1.5 inch gauge (distance between the rubber-coated jaws of the Instron Tester). The shorter sample is broken using zero gauge. The coherence value ( $C_h$ ) is the breaking strength of the longer sample divided by the breaking strength of the shorter sample. The average results from at least 3 pairs of such breaks in each direction of the fabric is reported as  $C_h$  in the Examples below.

For  $C_{h,wet}$  values, a bath of distilled water is raised around the sample which is clamped in the Instron machine and the sample soaked



for at least 1 minute before breaking it in the bath. All samples are elongated at a rate of 0.5 inch per minute.

- 5 Salt-free fabrics for use in the coherence value determinations are prepared by extraction with methanol water (60/40 by volume). About 100 g. of the fabric, divided into 4 portions (25 g. each) is placed in 1500 ml. of solution, then gently worked to insure saturation, soaked 15 minutes, and then gently squeezed and pressed between blotters. This is repeated using fresh solution until the solution is clear (normally 3 washes). Three additional treatments of the 4 portions simultaneously are then given followed by a final treatment in 100% methanol. The blotted samples are opened and dried in air. Twisting or pulling of the fabric during the treatments is avoided.

- 20 The *coefficient of sliding friction* ( $f$ ) (or kinetic friction) is the ratio of the force ( $F$ ) needed to pull one surface over another to the force ( $N$ ) normal to the surfaces in contact.

- 25 A fabric consisting of the fibre to be tested substantially free of salt (extracted to remove salt) of about 2 in.  $\times$  6 in. is placed on the horizontal bottom of a metal tank with the longer length in the direction of sliding and one end held in place with a heavy metal block.
- 30 A second portion of fabric of 1 in.  $\times$  3 in. is wrapped around a sled so that the bottom, front and back faces of the sled have 1 thickness of cloth and the top face has 1 to 2 thicknesses. A 5 gram brass weight is placed on top of the fabric. The sled consists of a 1  $\times$  1  $\times$  0.063 in. piece of poly (methyl methacrylate) plastic with an outer layer of crocus cloth (abrasive face out) (crocus cloth is an extremely fine abrasive cloth) held to the plastic with double-faced adhesive tape. The sled has a filament attached to each side near the forward edge forming a yoke. 0.1% Aqueous sodium chloride is slowly added to the tank to a depth of about 8 mm, the sled, fabric and weight placed on top of the lower fabric, the yoke attached to a filament leading under a pulley in the tank and upward to the crosshead of an Instron Tester and the tester started so as to pull the sled at a rate of
- 50 2 in. per minute. The sliding is continued for about 90 seconds. The charted force from 30 seconds to the end is averaged and reported as  $F$ . Force  $N$  is the sum of the sled weight, the fabric (normally about 0.15 g.) and the 5.0 g. weight, minus the buoyancy of the solution. The solution completely covers the sled and about 50% of the weight. (Total effective force,  $N$ , 5.6 gms.).

- 60 The above conditions should be followed since  $f$  varies with  $N$ .

- The test is made on a nonwoven fabric consisting of 100% of the fibres to be tested. Fabric pattern or structure variation has been found to have little effect on the sliding friction with essentially planar fabrics.

The *blending length* is half the length of a strip of sample that blends under its own weight to a 45° angle. It is determined on 1  $\times$  6 inch sample on a Drape-Flex Stiffness Tester (made by Fabric Development Tests, Brooklyn 32, N.Y.). The procedure utilized for the bending length determination is as described in American Standards of Testing Materials (ASTM) test number D 1388-55T. Unless otherwise stated the CD value is given.

The *fabric weights* are expressed in ounces/square yard, and are based on the weight of the fibres present minus water-soluble impurities.

The properties of the fabrics of the invention were determined by the methods discussed below.

The *dispersibility* is determined in a 250 ml. filter flask having an added side arm at the bottom of the conical wall and containing a magnetically rotated bar. The bar is 3.8 cm. long by 8 mm. in diameter, weighs 11.73 grams and is rotated at 500 revolutions per minute. The flask is filled with water. A 3  $\times$  3 inch sample is folded in half and inserted under the surface of the water (at the top side arm). Tap water at about 25°C. is added through the bottom tube at a rate of 0.70 litres/minute for 2 minutes. The effluent liquid from the upper side arm is filtered and the residue dried to constant weight at 100°C. to give the weight of fibres dispersed. The contents of the filter flask are filtered after the test and dried to yield the weight of undispersed fibres. The percent dispersibility is equal to 100 times the weight of fibres dispersed divided by the total weight of fibres recovered. Conventional toilet tissues have a dispersibility of 7%.

The *flushability* of a sample is determined by dropping a 10  $\times$  26 inch sample that has been folded to 10  $\times$  13 in. into the bowl of a household toilet (Model F2122 made by the American Radiator and Standard Sanitary Corporation of New York, New York) and flushing. The discharge from the toilet is passed through a length of horizontal glass pipe 2.33 ft. long and of 10.8 cm. inside diameter containing an artificial obstruction. The obstruction is constructed of standard flattened expanded metal with 0.5 in. wide perforations, formed in a cylinder of 1 ft. long and about 10.8 cm. in diameter and provided with 41 inside projections randomly distributed and made by making parallel pairs of cuts about 0.25 to 0.75 in. long and about 0.3 in. apart, and bending the cut sections to stand perpendicular to the walls of the cylinder. One flushing gives a flow of about 20 litres of water in 7 to 8 seconds. The toilet is flushed 3 times for each sample. The percentage of the sample that passes the hooks in the glass pipe is estimated and recorded after each flush. A sample is termed "flushable" if at least 60%

passes by the hooks after 2 flushes. At least 85% of a sample of the preferred products passes by the hooks after 2 flushes.

- 5 It has been observed that the samples of Examples I to VI termed "Flushable" by the above test have a dispersibility of at least about 20% (i.e., in 2 minutes), or require less than 20 minutes to disperse completely in the small-scale dispersion test. Preferred products  
10 have a dispersibility of at least 40%.

- The *loose fibre test* is a measurement of the fabric integrity and is conducted by rolling a wet sample of the nonwoven fabric (9.8 × 12 cm.) on a glass plate once with a dry rubber roller having a surface area equal to the sample. The fibres adhering to the roller are collected, washed with acetone, dried and weighed. Before testing, the sample is thoroughly wetted with an excess of distilled water, covered with 2 paper towels and blotted by rolling lightly once with the above roller.  
15  
20

- Nonwoven structures with no entanglement of the fibres lose about 0.12 gram by this test (or 14% of the original 0.86-gram sample). The preferred fabrics of this invention show zero loss by this test. Useful structures may show a loss as high as 8%. Fabrics having a loss of less than about 10% are deemed to be unitary.  
25

- The invention is illustrated by the following Examples wherein all solutions are aqueous unless otherwise stated. All screens are designated by the wires/inch and are understood to be the same in both directions, i.e. square mesh unless otherwise stated. The amount of liquid on a fabric is designated by the "pick up" which is the grams of liquid on the fabric per gram of original dry fabric.  
30  
35

#### EXAMPLE I

- 40 Cyanoethyl cellulose fibres of 0.4 degree of cyanoethyl substitution, 2 denier per fibre, and 0.5-inch staple length are prepared. A mixture of 80 parts of these fibres (based on the dry weight of the water-insoluble fibre) and 20  
45 parts of viscose rayon 0.25-inch staple of 1.5 dpf is added to water at 30°C. in a stock chest and well agitated to give a uniform slurry containing 0.24% fibres. This slurry is diluted to a consistency of 0.12% and fed to a headbox on a Fourdrinier machine as described previously. The machine wire is 31 inches wide made of 7.5-mil diameter wire in a 70 × 52 mesh/inch construction. The partially-drained fibre layer containing swollen  
50 cyanoethyl cellulose is sprayed with 20% sodium sulphate at 40°C. at 2, 3, and 4.75 feet from the breast roll. The nozzles supply about 1.3 gallons minute. The salt solution deswells the fibres. The wet paper is passed  
55 from the couching roll to a pressing section  
60

where it is pressed and is then dried on rolls heated to 120°C. The dry layer (A) has a fabric weight (water-insoluble fibre) of 1.54 oz./yd.<sup>2</sup>.

A second layer (B) with a fabric weight of 0.80 oz./yd.<sup>2</sup> is made from a mixture containing 10% of the above cyanoethyl cellulose staple and 90% of the rayon staple by the above procedure with omission of the salt solution spray. The stock chest consistency of 0.06% is reduced to 0.02% just prior to entering the headbox.  
65  
70

An assembly of layer (A) covered with layer (B) is continuously fed to a belt of a 24 × 24 wires/inch screen, woven of 20-mil diameter wire, moving at 6 feet per minute.  
75

The assembly (on the screen) is hydraulically entangled by streams of a 20% sodium sulphate at 50°C. from a single row of 5-mil diameter orifices spaced about 25 mils centre-to centre in a 12-mil thick plate mounted on a manifold. The orifices are spaced about 1 inch from the fibres and a pressure of 300 psi is maintained on the salt solution in the manifold. Both ends of the horizontal manifold are oscillated by eccentrics on a 0.5-inch diameter eccentricity at a rate of 9 to 10 revolutions/second. At the pressure used, the 27-inch long row of orifices delivers 165 pounds of salt solution per minute through the 1080 orifices onto the 27-inch wide layer of fibres. The total weight of the double layer of fibres passing under the fluid streams in 1 minute is 0.220 pounds. The Y value is thus  $11 \times 10^6$  feet/minute.  
80  
85  
90  
95

If the salt solution is replaced with water alone, the fibres are washed away and/or driven partially through the screen.

The unitary nonwoven fabric produced is pressed between blotting papers at 1450 psi. Portions of the pressed fabrics (about 50—60% solids) of about 10 × 26 inch are dried and softened by tumbling in a home laundry dryer at about 45°C. with 4 baseballs of 9.7 cm. in diameter.  
100  
105

Drying without softening yields a web that is stiff, board-like, has poor drape and no aesthetic appeal.

The dry, softened fabric is a unitary nonwoven fabric with the 2 layers intimately connected. It has the appearance and hand of a soft woven fabric. Figure 3 is a photograph at 10X magnification of the face of the fabric that faced the liquid streams.  
110

The softened nonwoven fabric has a fabric weight of 2.6 oz./yd.<sup>2</sup> contains 12.5% of sodium sulphate, 41.1% cyanoethyl cellulose and 46.4% of rayon and has a density of 0.138 g./cm.<sup>3</sup>. Properties (corrected to a salt-free basis) of the fabric are given below.  
115  
120

Property	Non-softened		Softened	
	MD	CD	MD	CD
Tensile, dry lb./in.	4.43	1.37	2.57	1.64
Elongation, dry (%)	5	38	28	27
Tensile strength, wet lb./in.	0.039	0.028	0.030	0.027
Elongation, wet (%)	31	36	26	26
Bending length (cm.)	3.8	2.8	2.0	1.4
Dispersibility, (%)	48		54	
Flushability, 1st flush	50		100	
2nd flush	100		—	
Coherence value — dry/wet			0.46/0.08	
Coefficient of sliding friction (f wet) of the cyanoethyl fibres			0.48	

For use as a diaper the nonwoven fabric is folded to give a double thickness with the rayon-rich faces on the outside. The diaper is pinned about the baby in the usual manner. The diaper will absorb the total urine output of an average 6-months old baby for a period of 4 hours (i.e., 75 grams of urine). Even under this extreme condition the diaper retains its integrity, does not tear at the pins, leaves no loose fibres on the skin and is non-irritating. The soiled diaper is dropped into a toilet and flushed away. This product is completely biodegradable so that no problems with sewage disposal are caused.

The nonwoven fabric may be used as a water-flushable sanitary garment and has a tensile strength in synthetic urine of greater than 0.05 lb/in.

#### EXAMPLE II

This Example shows the effects of variables in the hydraulic entangling step.

Two single layers of fibres are made from cyanoethyl cellulose fibres and viscose rayon staple of Example I by the method of Ex-

ample I. One layer contains 70% of the cyanoethyl cellulose fibres and has a nominal fabric weight of 1.18 oz./yd.<sup>2</sup>. The other layer contains 10% of the cyanoethyl cellulose fibres and has a nominal fabric weight of 0.77 oz./yd.<sup>2</sup>.

A series of nonwoven fabrics are made by hydraulically entangling a top layer of the rayon-rich fibres to a layer of the cyanoethyl cellulose-rich fibres under various conditions. Each of the products has an average composition of 46.5% cyanoethyl cellulose fibres.

Items (a) to (i) are prepared with an orifice assembly having 540 orifices of 5-mil diameter evenly spaced in a single 27-inch row. Items (j) to (m) are prepared with an orifice assembly having 1080 of the orifices of 5-mil diameter in a single 27-inch row. The same patterning screen and general conditions of Example I are used for both series, but pressures from 200 to 500 psi. are used to give different total flow rates (W) and the rate of traverse (V<sub>r</sub>) is varied. The entangling conditions and properties of the products are given in Table I.

TABLE I

Item	Hydraulic Entangling Conditions			Coherence Value		Tensile strength, dry	Tensile Strength Wet, Average	Elongation Dry	Flush- ability	Dispers- ibility
	$W$ lb/min	$V_s$ (ft/min)	$Y$ (ft/min $\times 10^6$ )	$C_h$ dry	$C_h$ wet					
a	79	6	6.5	0.2	0.13	0.89/0.72	0.016	25/14	100	54
b	79	5	7.6	0.46	0.06	0.68/1.65	0.027	32/16	100	50
c	79	4	9.3	0.39	0.06	1.5/0.98	0.027	40/21	100	48
d	79	3	12.5	0.20	0.07	1.4/0.35	0.032	30/26	100	46
e	101	6	10.2	>0.2	0.12	1.5/0.86	0.030	34/29	100	48
f	101	5	12.5	0.27	0.11	1.3/1.4	0.033	28/23	—	40
g	101	4	15.7	>0.2	<0.15	2.1/1.6	0.041	46/33	100	63
h	145	6	20.7			2.3/1.4	0.12	44/29	50	24
i	145	3	42.0	**0.30	0.49	3.3/1.3	0.21	37/32	55	8
j	137	5	11.1	0.24	0.05	1.1/1.0	0.039	33/27	100	50
k	166	6	13.6	0.44	0.10	2.0/1.2	0.090	36/28	90	32
l	195	4	28	0.55	0.44	2.6/1.5	0.27	40.37	40	11
m	186	3	35	**0.49	0.74	3.4/2.1	0.33	45/26	50	21

\*\* Estimated



- Items (a) to (g), (i) and (k) are fabrics of the invention with dry strip-tensile strengths sufficient for use demands (such as pinning), wet strip-tensile strengths low enough to give ready flushability but high enough in body fluids to be useful, blending lengths of 1.1 to 1.8 cm. and a soft handle. These products have densities of 0.113 to 0.150 g./cm.<sup>3</sup>
- based on thicknesses measured with an Ames gauge.
- EXAMPLE III**
- The fibres of Example 1 are used to make a composite sheet having two layers of fibres as described below, using a double-layer head-box and the method of Example I.

Fibre layer	CNEC %	Rayon %	Fabric Weight oz./yd. <sup>2</sup>
Bottom	10	90	0.9
Top	100	0	1.3
Average	64	36	2.2

- Portions of the sheet are treated with fluid streams of 20% sodium sulphate at various pressures and speeds by the method of Example I but with an orifice assembly having 280 orifices of 5-mil diameter evenly spaced in a single row 14 inches long. The apertured patterning member is a 20 × 20 wires/inch screen of 0.025-inch diameter wire. The rayon-rich face of the sheet faces the fluid streams. Pressures range from 50 to 500 psi. The rate of traverse ( $V_t$ ) is 6 feet per minute for items (a), (b) and (c) and 2 feet per minute for item (d). The total flow rate ( $W$ ) of the fluid streams and the calculated  $Y$  values are given in Table II, as are the properties of the fabrics.

TABLE II

Item	Entangling Conditions		Coherence Value C <sub>h</sub> dry/wet	Tensile strength, dry	Elongation dry	Tensile Strength Wet, Average		Dispersibility %
	W lb./min.	Y ft./min. × 10 <sup>6</sup>		lb./in. MD/CD	% MD/CD	lb/in.		
a	24	1.9	>0.2/<0.15	4.0/0.88	9/3	0.0107	69	
b	30	2.9	>0.2/	4.8/0.49	11/19	0.0027	79	
c	53	9.1	0.65/0.08	3.4/2.6	28/28	0.025	67	
d	76	55	0.56/0.61	4.8/2.3	30/43	0.23	8	

All fabrics have C<sub>h</sub> dry >0.2 and items a, b and c have C<sub>h</sub> wet <0.15 and a bending length of less than 3 cm.

All water-sensitive fibres have f (wet) of <0.70.

Item (c) represents a preferred fabric of this invention. It has the appearance, handle and elongation of a fabric. It does not lose fibres in the loose-fibre test.

Item (a) has the appearance of the original untreated sheet, with essentially no patterning or even embossing. More than about 8% of its weight is lost in the loose fibre test.

Item (b) has some resemblance to a fabric but the pattern is not so distinct and uniform as that of item (c). The product loses about 8% of its original dry weight in the loose fibre test.

Item (d) has the appearance and handle of a fabric but the fibres have been so firmly entangled that it is not flushable.

Items (a)---(d) have a bending length less than 3 cm.

EXAMPLE IV

This Example illustrates the use of highly water-absorbent fibres of different types of cellulose ethers in preparing the products.

Hydroxyethyl cellulose (HEC) fibres were prepared as follows.

Conventional wood pulp sheets are steeped in 18% caustic soda at 27.5°C. for 45 minutes. The sheets of alkali cellulose are pressed to a 3/1 press weight ratio, shredded, and stored at 0°C.

One pound of the alkali cellulose is held at 26°C. for 27 hours, is added to a 5-litre churn and the air is evacuated. Fifty-three grams of ethylene oxide are added over a 90-minute period as it is revolved on its side in a water bath at 25°C. The churn is flushed with nitrogen and the hydroxyethyl cellulose pro-

- duced is removed and dissolved in dilute sodium hydroxide at 0° to 10°C. to give a solution containing 6% of the cellulose ether and 7% caustic. The solution is frozen at -30°C., heated to room temperature and filtered. The filtered solution is extruded through a spinnerette containing 504 orifices of 3-mil diameter into a coagulating bath (12% sulphuric acid and 18% sodium sulphate) and the yarn is neutralized. The dry yarn has a dpf of 2.6, a dry tenacity of 0.98 gpd, a dry elongation of 9.8% (all reduced to a salt-free basis), respectively, and a degree of hydroxyethyl substitution of 0.61.
- 15 Cyanoethyl cellulose fibres are prepared as for Example I except that the modified viscose solution is aged for 3 days at 25°C., and then spun and neutralized as previously described. The fibres are washed with 70°C. distilled water, deswollen with acetone and dried at 100°C. The fibre has a degree of substitution of carboxyethyl groups of 0.13 and a degree of substitution of cyanoethyl groups of 0.04.
- 20 Cyanoethyl cellulose fibre (CNEC) were also prepared by a different route from Example I. A solution of cyanoethyl cellulose (CNEC) is prepared according to British Patent Specification No. 633,807 by adding the acrylonitrile to conventional viscose. The solution is spun to fibrous and neutralized as described previously. The fibres have the following typical properties calculated on a water insoluble fibre weight:
- 35 Denier 2  
Water absorbency (25°C.) 19.1 g./g.  
Urine absorbency (25°C.) 14.0 g./g.  
% Nitrogen 3.4  
% Carboxyl 0.4  
40 Degree of cyanoethyl substitution 0.45  
Degree of carboxyethyl substitution 0.015

Each of the above cellulose ether fibres is cut to 0.5-inch staple length and handsheets are prepared from aqueous slurries of the fibres in an 8 × 8-inch sheet mould. The handsheet and the sheet mould screen are removed from the sheet mould box, the fibres are deswelled by wetting with 18% sodium sulphate for about one minute, and the sheet is then dried. The following sheets are prepared in this manner, the weights and percentages being based on the water-insoluble fibre content:

- Sheet (c), 1.43 oz./yd.<sup>2</sup>, 100% CbEC and 61% of 0.25-inch viscose rayon fibres; 55  
Sheet (b), 1.43 oz./yd.<sup>2</sup>, 100% HEC fibres; Sheet (c), 1.43 oz./yd.<sup>2</sup>, 100% CbEC fibres; and  
Sheet (d), 1.43 oz./yd.<sup>2</sup>, 100% CNEC fibres. 60

Sheet (a) is hydraulically entangled with essentially columnar streams of 20% sodium sulphate as in Example I. Sheet (b) is plied with a layer B of Example I, containing 10% cyanoethylcellulose fibres and 90% rayon fibres, and the assembly is hydraulically entangled, as above, to form a composite (b-B). Composites (c-B) and (d-B) are similarly prepared. The treatments were varied in the range of Y values from  $6.6 \times 10^6$  to  $20 \times 10^6$  to give different structural properties.

The products are dried and softened as in Example I. All have dry-strip-tensile strengths of at least 1.8 lbs. inch and dry elongations of at least 38%. Other properties are given in Table III.

All fabrics have ( $C_h$  dry) >0.2 and ( $C_h$  wet) <0.15 and a bending length of less than 3 cm. All water sensitive fibres have f(wet) of >0.70.

TABLE III

Item	Average Composition	Tenacity, Wet (g./cm.) lb/in.	Elong. dry %	Dispersibility %
a	39% HEC	0.012	38	100
b-B	64% HEC	0.048	46	74
c-B	64% CbEC		55	40
d-B	64% CNEC	0.063	52	56

## EXAMPLE V

5 Cyanoethyl cellulose (CNEC) fibres of 0.5 inch length and less than 5 dpf, and containing 43.9% of sodium sulphate and analyzing at 3.5% N (salt-free basis) and 0.5% COOH (salt-free basis) with a D.S. (cyanoethyl) of 0.465 and 0.25 inch length rayon staple of 1.5 dpf are used as fibres. The (CNEC) fibres have an  $f$  (wet)  $< 0.70$ .

10 Handsheets of different compositions and a fabric weight (salt-free) of 1.5 oz./yd.<sup>2</sup> are made from aqueous slurries of the above fibres. The sheets are deswollen in 20% sodium sulphate, pressed between couch rolls and dried between nylon fabric and blotting paper on a sheet dryer at 100°C.

20 All sheets are hydraulically entangled by passing the sheet resting on a 24 × 24 wires/inch screen at 3 feet minute under oscillating streams of 20% sodium sulphate. Water is used as the entangling fluid for the 100% rayon sheets. The streams are produced by a plate containing a single row (14" long) of 0.007 inch diameter venturi shape, orifices spaced 20/inch, which is mounted on a manifold. The orifice plate is located about 1 inch above the handsheet.

30 The entangled sheets are blotted and then dried and softened by tumbling in a home laundry dryer at 60°C. with 3 baseballs of 9.7 cm. in diameter for 25 minutes.

The physical properties obtained at various Y values (by altering the pressure of the entangling fluid) are given in Table IV. All

samples have dry elongations of from 20 to 56% and a bending length of  $< 3$  cm. 35

Items a, b and c, represent unsuccessful attempts to make useful flushable fabrics of 100% rayon. Water pressures of 100, 150, and 200 psi are used. The combination of a dry tensile strength of at least 0.3 lb./in. and a wet tensile strength of less than 0.1 lb./in. is not obtained. At the higher Y values needed to increase the dry strength, the fabric is not flushable due to the higher wet strength obtained and lower dispersibility. 40 45

Items f, h, j, l, m, n, o and p represent fabrics of this invention, and have  $C_h$  (dry)  $> 0.2$  and  $C_h$  (wet)  $< 0.15$ .

Items g, i and k have been excessively entangled to give 0.1 lb./in. and dispersibilities of 15 and less so that they are not flushable. Thus, they are not fabrics of the present invention. 50

It is seen that the hydraulic entangling conditions are extremely critical, particularly at the lowest cyanoethyl cellulose levels. The results in the Table and results at other (unreported) Y values can be expressed by the following equation: 55 60

$$Y_{\text{maximum}} = 11 + 1.4 (\% \text{ cellulose ether})^2$$

Thus the maximum Y values for 10 and 100% of cellulose ethers are about 15 and 25, respectively. This relationship applies only to the preferred blends of cyanoethyl cellulose with rayon. 65



TABLE IV

Item	$Y \times 10^{-6}$	% CNEC	Tensile Strength, lbs./in.		Dispersibility %	Fabric Weight oz/yd. <sup>2</sup>
			dry	wet		
a	7.0	0	0.12	0.088	32	0.92
b	10.8	0	0.16	0.137	16	0.97
c	14.6	0			5	1.07
d	6.8	5	0.33	0.047	48	1.17
e	7.9	5	0.41	0.057	38	1.07
f	12.4	10	0.62	0.061	48	1.60
g	19.1	10	1.43	0.13	6	1.60
h	12.4	15	0.58	0.065	39	1.49
i	19.1	15	1.01	0.158	8	1.14
j	12.4	20	0.71	0.090	54	1.60
k	19.1	20	1.29	0.113	7	1.38
l	12.4	50	1.65	0.052	58	2.18
m	19.1	50	1.0	0.079	31	2.18
n	12.4	75	2.28	0.045	62	2.64
o	19.1	75	0.95	0.056	29	2.64
p	12.4	100	1.96	0.062	56	2.74

## EXAMPLE VI

This example illustrates the use of a non-cellulosic high-absorbency fibre in preparing a soft, water-dispersible, nonwoven fabric.

5 A solution of 99% hydrolyzed, high-viscosity polyvinyl alcohol in dimethyl sulphoxide is extruded through a spinnerette into a high-temperature evaporative atmosphere by methods well known in the art. The dried  
10 filaments are wound up and later drawn 1.5 times, crimped in a stuffer box and cut to 1/4 inch staple. The polyvinyl alcohol fibre has an absorbency of 13 g./g. in water at 37°C. and a urine absorbency of 9.6 g./g.

15 The polyvinyl alcohol staple fibres are mixed with an equal weight of 1/4 inch viscose rayon staple. The mixed fibres are slurried in ice water and formed into a sheet on a Noble and Wood hand sheet mould. The  
20 highly-swollen sheet is covered with a protective screen and deswelled in 20% sodium sulphate.

The wet deswelled web of mixed fibres is transferred to a 24 × 24 wires/inch screen woven of 20-mil diameter wire. The fibres are hydraulically entangled with streams of 20% sodium sulphate at 50°C., delivered at 200 psi pressure to an orifice assembly having 560 orifices of 5-mil diameter evenly spaced in a  
30 14-inch row. The wet nonwoven fabric is pressed between bleached sulphite blotters and dried by tumbling 15 minutes at 32°C. in a laundry dryer with one baseball of 9.7 cm. in diameter. The final soft dry nonwoven  
35 fabric has the following properties:

	Fabric weight (oz./yd. <sup>2</sup> , insoluble fibre)	1.89
	Tensile, dry, lbs./in.	0.34
	Elongation, dry, %	62
	Tensile, wet, lb./in.	0.028
40	Elongation, wet, %	34
	Dispersibility, %	49
	Bending length, cm.	1.18
	C <sub>h</sub> , wet	0.1
	S <sub>h</sub> dry	>0.2
45	f (wet)	<0.70

## EXAMPLE VII

A nonwoven fabric of 1.2 oz./yd.<sup>2</sup> weight is made by passing a random web of 0.75 in. rayon fibre of 1.5 dpf on a 24 wires/inch screen, at 5 yards per minute under 3 rows of water streams coming from orifices having an upper cylindrical section of 0.005 inch diameter with a lower frusto-conical section as an exit and spaced 40 per inch at pressures of  
50 400, 500, and 600 psi. The orifices are about 12 mm above the web. The rayon fabric has a C<sub>h</sub>, dry and C<sub>h</sub>, wet of 0.35 and 0.71 respectively. The fabric at this point is not a fabric according to the present invention.

60 The nonwoven rayon fabric is passed through a tank containing a solution of 50% urea and 18% phosphoric acid at room temperature. The fabric is squeezed by a pair of

rubber-covered rolls to a pickup of about 2.0 g./g. The wet fabric is continuously passed  
65 through a commercial fabric oven (Benz) where it is exposed to opposed jets of air at 164°C. for a period of about 91 seconds and then wound up at about 6.3 feet per minute.

The baked fabric (cellulose acid phosphate) is then carried on a screen underneath sprays of tap water for washing and excess water is removed by squeeze rolls. The wet fabric is then carried under sprays of a solution containing 17% sodium sulphate, 3% disodium hydrogen phosphate and 1% sodium hexametaphosphate adjusted to a pH of 6.0 and excess solution is removed by squeeze rolls.

The wet, modified fabric (sodium cellulose phosphate) is then passed through an oven  
80 where it is exposed to air at 34–40°C. issuing from pairs of opposed slots transverse to the direction of fabric so that the fabric flutters between the 2 opposed jets of air. The dry fabric is then softened by passing between a  
85 pair of meshing grooved rolls with the grooves running in the direction of travel of the fabric followed by 3 pairs of meshing groove rolls having grooves transverse to the direction of travel of the fabric. All of the rolls are set at  
90 3.6 lb/inch of width pressure. The softening treatment is repeated two more times. C<sub>h</sub> dry is 0.29 and C<sub>h</sub> wet 0.04.

Diaper size portions of the product are flushable. The properties are given in Table V. 95

## EXAMPLE VIII

This Example illustrates the use of cellulose sodium hemisuccinate in this invention.

The nonwoven rayon fabric of Example VII is passed through a tank containing a solution at 60°C. of 30% succinamic acid and 0.60% sulphamic acid, and squeezed by rolls to a pickup of 2.0–2.1. The wet fabric is continuously passed through a circulating air oven (205°C.) at a speed to give a residence  
100 time of about 95 seconds.

The fabric of cellulose hemisuccinate is washed by passing through a tank of soft water, squeezed and then neutralized and bleached by passing through a tank containing a solution at room temperature of 3% disodium hydrogen phosphate, 17% sodium sulphate and about 0.5% potassium peroxy-monosulphate, adjusted to a pH of about 8.5–9.0. The fabric of cellulose sodium hemisuccinate is squeezed and dried in circulating air at room temperature.

Diapers made of the product are tested on babies and found to have excellent integrity in use and the used diapers are completely  
110 flushable. Properties of the product are given in Table V.

## EXAMPLE IX

This Example illustrates the use of fibres of sodium cellulose sulphate in the inven-  
125 tion.

A nonwoven fabric of 1.3 oz./yd.<sup>2</sup> weight is

made from a random web of 0.75 inch crimped, rayon fibres of 1.5 dpf using the apparatus and speed of Example VII at pressures of 300, 500, 800 and 800 psi for the 4 rows of orifices.

- 5 The rayon fabric is placed for 2 minutes in a solution at 50° of 300 g. urea, 100 g. 99% sulphuric acid and 75 g. of water. It is then pressed to a pickup of 3.2 and the wet fabric heated in an oven at about 169°C. for 3 minutes. The heated fabric is neutralized in a solution containing 17% sodium sulphate and 3% disodium hydrogen phosphate, adjusted to a pH of 8.5, squeezed to a pick-up of 1 g./g. and dried. The product is flushable. Properties are given in Table V.

#### EXAMPLE X

- 20 This Example shows the use of sodium carboxymethyl cellulose fibres in this invention.

- 25 A nonwoven fabric of 0.8 oz./yd.<sup>2</sup> weight is made from 1.56 inch rayon fibres of 1.5 dpf using the procedure and equipment of Example VII except that 2 additional rows of orifices at 200 and 300 psi pressure are used. The fabric has a C<sub>h</sub>,dry and a C<sub>h</sub>,wet of 0.74 and 0.96 respectively.

- 30 The fabric is chemically modified by passing it continuously through a bath containing 2.1% sodium hydroxide and 15.8% sodium chloracetate, squeezing it to a pickup of 1.1 g/g heating in an oven with air at 140°C. for 18 seconds, acidifying to the carboxymethyl cellulose in a bath of 5% sulphuric acid, washing in water and forming the sodium salt in a bath of 2% disodium hydrogen phosphate and 8.5% sodium sulphate, adjusted to a pH of 8.5. The fabric (A) is dried at 40°C. in a tumble dryer with baseballs of 9.7 cm. diameter.

- 40 Sanitary napkins are made by wrapping a 19" × 7" portion of the fabric around a core, overlapping the fabric on the bottom of the napkin and gluing the overlap with small spots of a water soluble adhesive. The core consists of wood fluff wrapped with crepe tissue paper which contains a coating of a poly(fluoro alkyl methacrylate) on the sides and bottom of the paper to serve as a fluid barrier. The napkins are used and then flushed. The vast majority (223 out of 230) of the napkins flush with 1 rush of water. The other 7 require a second flush to clear the bowl.

- 55 A second fabric (B) of 1.25 oz./yd.<sup>2</sup> having a C<sub>h</sub>, dry and C<sub>h</sub>, wet of 0.66 and 0.78, respectively, is made as above except that a speed of 7 yards per minute under jets at 300, 500, 600, 700 and 900 psi is used. The fabric is chemically modified as above except that the reagent solution contains 2.22% sodium hydroxide and 18.4% sodium chloracetate and the final salt-forming solution contains 1% disodium hydrogen phosphate

and 4.25% sodium sulphate, adjusted to a pH of 8.5. 65

Diapers are assembled with a layer of the above fabric, a wood fluff, a crepe tissue wadding, wood fluff and the above fabric, in that order. A medium size diaper uses 11 × 14" fabric and 5 × 12" cores of wood fluff and crepe tissue. The 2 fabric layers are glued together around the edges of the core with about 3 mm spots of a water-soluble adhesive spaced about 5 cm apart. 70 75

The diapers are used on babies in wear tests and are found to have good integrity in use and all are successfully flushed in home toilets after use.

Properties of the fabrics are given in Table V. 80

#### EXAMPLE XI

This Example shows the preparation of a non-patterned fabric of the invention.

A nonwoven fabric of 2.3 oz./yd.<sup>2</sup> is made from the rayon fibre of Example VII-, using the general method and a single row of orifices of that Example, with a 150 mesh per inch screen (37% open area) used as the support to carry the web at 4 yards per minute under the oscillating row of orifices for one pass at each of the pressures of 200, 500, 1000, and 1200 psi. The nonforaminous fabric is smooth and resembles a felt with faint grooves. 85 90 95

A portion of the dry fabric is immersed in a solution of 50% urea and 18% phosphoric acid for 2 minutes, drained and blotted to a pickup of 1.6—1.7. The wet fabric is heated in an air oven at 160°C. for 4 minutes. The fabric is then washed in an aqueous solution of 5% sulphuric acid and 17% sodium sulphate, drained, blotted, and converted to the sodium salt of cellulose phosphate in an aqueous solution of 4% disodium hydrogen phosphate and 17% sodium sulphate, adjusted to a pH of 8.4. The fabric is drained, blotted, and air dried. 100 105

A medium size diaper is prepared. The diaper breaks into 2 portions after dipping it ten times in a toilet bowl and 85% of the diaper passes the hooks after 3 flushes. Properties of the product are given in Table V. 110

#### EXAMPLE XII

This Example shows the use of a heterogeneous water-sensitive fibre in the invention. 115

A nonwoven fabric of 0.8 oz./yd.<sup>2</sup> weight is made from a random web of 0.5" rayon of 1.5 dpf using the apparatus and method of Example VII but with 4 rows of orifices at 300, 400, 500 and 500 psi. 120

The dry fabric is placed in a solution of 70 volumes of water and 30 volumes of N-dimethyl formamide for 1 minute and is then squeezed to a pickup of 1 g/g. The wet fabric is then placed in a 0.25N solution of sulphur 125

trioxide in N-dimethylformamide at room temperature for 1 minute and squeezed to a pickup of 1 g/g. The fabric is then treated in a 5% aqueous solution of sulphuric acid for 1 minute, squeezed and then treated in an aqueous solution containing 17% sodium sulphate and 3% disodium hydrogen phosphate, adjusted to a pH of 8.5 for 1 minute, squeezed and air dried to yield a fibre of cellulose (rayon) with an outer skin of the sodium salt of cellulose sulphate.

A 17 x 13 inch portion of the fabric is soaked in synthetic urine and dipped 2 times in water; all the fabric can be flushed away. The fabric has the properties as given in Table V. The average D.S. of the rayon core and the modified skin is 0.008.

#### EXAMPLE XIII

The Example illustrates the greater internal bond strength of the fabrics of this invention over the prior art structures.

The internal bond test, originally designed to measure the internal bond strength of

papers, is useful as a measure of the vertical entangling component through the thickness of the fabric.

In essence, the test consists of measuring the force necessary to rupture the test specimen when the plane surfaces are mounted between and held by two pieces of adhesive tape.

As expected, a laminar type structure is readily parted (low internal bond) whereas a structure held together by a vertical component requires higher forces since the vertical fibers must be broken.

The *Internal Bond Strength* of a nonwoven fabric is determined on an Internal Bond Tester (model B — made by Sisalkraft-Scott Co. of Providence, R.I.) by measuring the force required to shear a sample in its major plane with a weighted pendulum. The sample is fastened to a lower fixed steel plate and to an upper aluminum right angle striking bar (which contacts the pendulum) by means of double-faced sensitive tape (Scotch Brand type 400 made by 3M Co. of St. Paul, Minn.).



TABLE V

Example	D.S.	Coherence Value		Tensile Strength, MD/CD, lb./in.		Elongation Dry MD/CD %	Fabric Weight oz./yd. <sup>2</sup>	f, wet
		C <sub>h</sub> , dry	C <sub>h</sub> , wet	dry	wet			
VII	0.26—0.30	0.29	0.04	6.1/2.6	0.018/0.011**	12/72	2.3	0.72
VIII	0.30	0.42	0.04	4.2/3.0	0.017/0.11	27/44	1.5	<0.70
IX	0.28	0.59	0.27	2.7/2.0	0.060/0.027	32/36	1.5	<0.70
X—(a)		0.66	0.11	4.1/2.8	0.0016/0.0015	28/77	1.2	<0.70
X—(b)		0.77	0.10	6.7/3.0	0.016/0.005**	20/77	1.8	0.31
XI	0.17	0.69	0.14	c. 6.8/9.1	c. 0.0010/0.0015	39/28	3.2	<0.70
XII		0.32	0.06	c. 2.4/1.0	c. 0.004/0.004	32/36	1.0	<0.70

c. indicates that the tensile strength was determined from the breaking force obtained in the coherence value test.

\*\* determined in tap-water.

Bending length for all the above fabrics is less than 3 cm.

5 The faces of the steel plate, striking bar, non-woven fabric and tape are 1 inch X 1 inch. The tester includes a jig for mounting five samples at a time at a standard pressure. Five samples are tested in both machine and cross direction and the average value reported. The nonwoven fabric should be in a salt-free state as obtained by extraction for the Coherence Value (C<sub>h</sub>) determination. Presures of 200 psi are used, although lower pressures may be required with very thin and porous samples to prevent forcing the upper tape into contact with the lower tape. Meaningful results are not obtained with coarsely apertured products [i.e. 12 or less apertures or mesh per inch with a fabric weight of less than about 1 oz./yd.<sup>2</sup> due to the adhesion of tape to tape through the thickness of the fabric.

20 Fabrics from a number of the previous Examples are tested as well as papers and a resin-bonded commercially available non-woven.

25 The preferred fabrics of this invention have internal bond strengths of at least 0.08 foot-pounds (ft.-lb.) while the more preferred fabrics having the most dry integrity, have internal bond strengths of at least 0.15 ft.-lbs.

Typical results are given below:

<i>Fabric from Example:</i>		<i>Internal Bond Strength</i>
		ft.-lbs
5	I, softened	0.26
	II, item d	0.09
	II, item 1	0.17
	VII	0.11
	VIII	0.19
10	XII	0.32
	typical rayon non-wovens used for example	0.25 to >0.5
	commercial, resin-bonded, rayon nonwovens	0.05 — 0.07
15	— Binder in about 1/16 in. bands on 1/4 in. centres across width for strength — papers, used as starting material for Examples 1 and 2	0.01 to 0.05

#### 20 WHAT WE CLAIM IS:—

1. An absorbent non-woven fabric of textile fibres secured in place by random fibre entanglement to provide dry strength and integrity in the absence of resinous or fusion bonds, the fabric comprising from 10 to 100% water-sensitive fibres (as defined herein) of less than three inches in length, the balance of the fibres consisting of non-water-sensitive fibres of less than 0.5 inch in length, the fabric having a  $C_{h \text{ dry}}$  value (as defined herein) of at least 0.2 and a  $C_{h \text{ wet}}$  value (as defined herein) less than 0.3, and the ratio of  $C_{h \text{ dry}}$  to  $C_{h \text{ wet}}$  being  $\geq 1.3$ .

2. A fabric according to claim 1 having a weight of 0.3—5 oz./yd.<sup>2</sup>.

3. A fabric according to claim 1 or 2 having an average wet strip tensile strength of less than 0.1 lb./in.

4. A fabric according to any one of the preceding claims wherein the fabric has an elongation at break in two directions at right angles to each other of at least 20%.

5. A fabric according to any one of the preceding claims wherein the  $C_{h \text{ wet}}$  value is less than 0.15.

6. A value according to any one of the preceding claims containing at least 40% of the water-sensitive fibres.

7. A fabric according to any one of the preceding claims having an average tensile strength in synthetic urine of at least 0.05 lbs./in.

8. A fabric according to any one of the preceding claims wherein the  $C_{h \text{ dry}}$  value is at least 0.3, the  $C_{h \text{ wet}}$  value is not more than 0.15 and the ratio

$$\frac{C_{h \text{ dry}}}{C_{h \text{ wet}}} \text{ is at least 2.}$$

9. A fabric according to any one of the pre-

ceding claims wherein the water-sensitive fibres have a denier per filament of 0.5 to 15 and a length of 0.25 to 2 inches.

10. A fabric according to any one of the preceding claims wherein the water-sensitive fibres are chemically modified cellulosic fibres comprising cellulose esters or ethers whose A—OH compound (as defined herein) is soluble in water at 20°C. to the extent of at least 3%.

11. A fabric according to any one of the preceding claims consisting substantially of water-sensitive fibres.

12. A fabric according to any one of claims 1 to 10 comprising 30—90% of water-sensitive cellulosic fibres and 70—10% of non-water-sensitive fibres, the fabric having a surface layer containing at least 80% of the non-water-sensitive fibres and a centre containing at least 70% of the water-sensitive fibres.

13. A fabric according to claim 1 substantially as described in any one of the Examples.

14. A process for preparing a fabric according to any one of the preceding claims, the process comprising supporting a non-woven web comprising at least 10% of fibres which are water-sensitive or are capable of being rendered water-sensitive, and are less than 3 inches in length, the remainder of the fibres being non-water-sensitive and not more than 0.5 inch in length, on an apertured member, directing fine essentially columnar streams of a liquid essentially inert to the fibres at an energy flux of at least 23,000 ft.-poundals/in.<sup>2</sup>-sec. at the web so as to impinge thereon, and moving the web relative to the streams, with the proviso that when the fibres in the web are water-sensitive before directing the stream thereat, the stream comprises a de-swelling liquid for water-sensitive fibres and has a Y value (as defined herein) of  $3 \times 10^6$  to  $25 \times 10^6$  ft./min.; and then if necessary rendering water-sensitive the fibres capable of being so rendered.

15. A process according to claim 14 wherein the fibres are rendered water-sensitive by applying to the fabric a 17—30% solution of an amic acid (as defined herein) in an amount 100—300% by weight of dry fibres, and heating the fabric at 165° to 210°C.

16. A process according to claim 14 wherein the water-sensitive fibres are rendered water-sensitive by applying to the fabric phosphoric acid and urea (in a ratio of 1:3 to 1:2) in an amount of 60—160% by weight of dry fibres, and heating the fabric at 125° to 210°C.

17. A process according to claim 14 substantially as described herein.

18. A fabric prepared by a process according to any one of the preceding claims.

19. A sanitary napkin comprising an absorbent core and a covering of a fabric according to any one of claims 1 to 13 and 18.

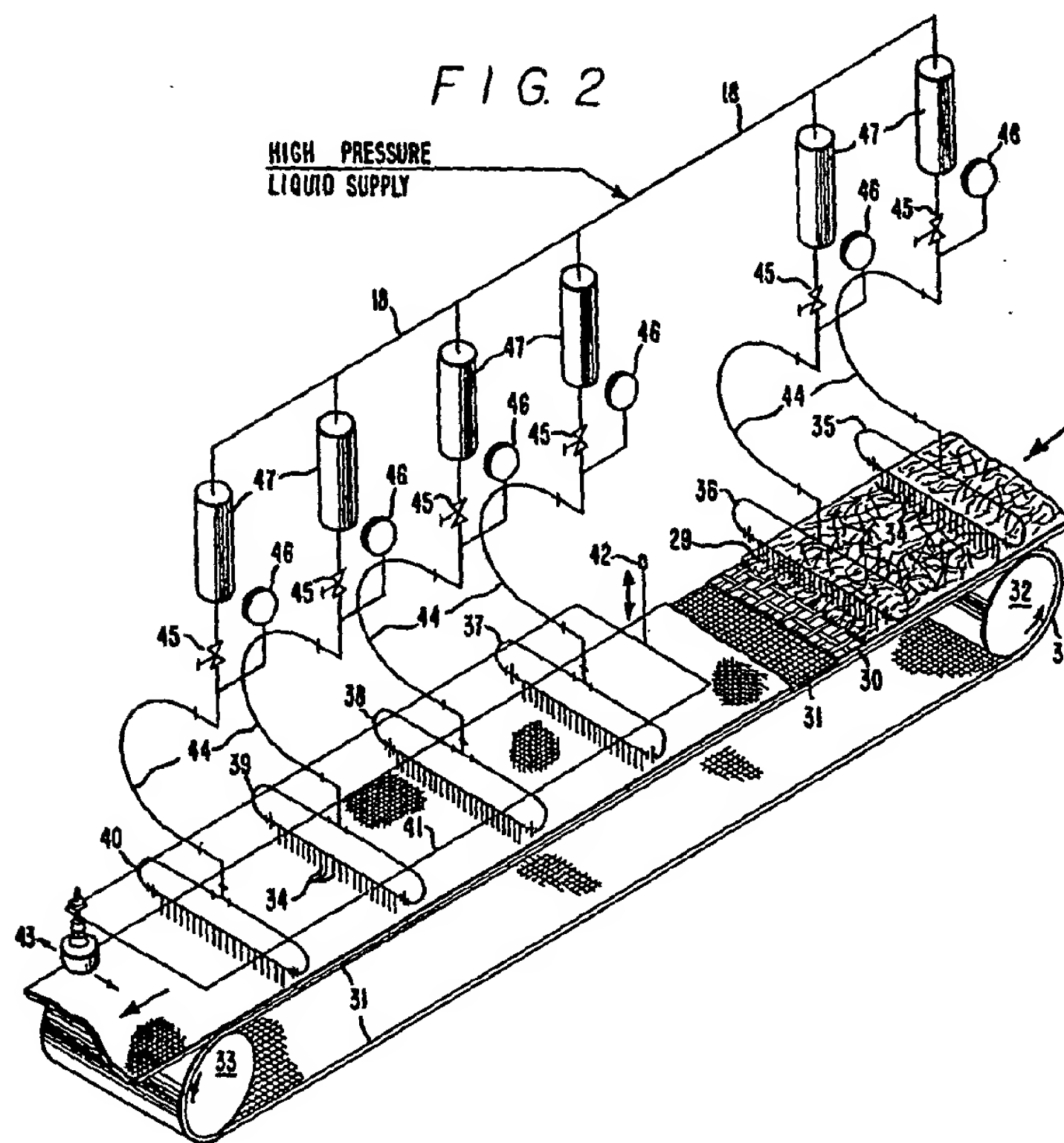
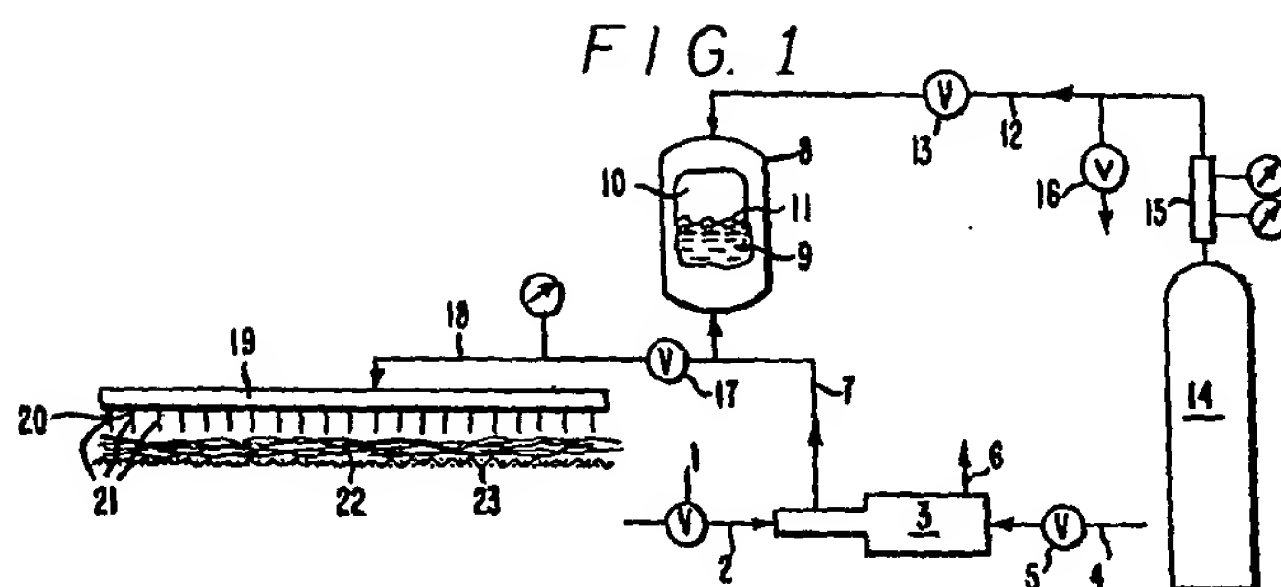
20. A diaper comprising an absorbent core

sandwiched between two layers of a fabric according to any one of claims 1 to 13 and 18.

J. A. KEMP & CO.,  
Chartered Patent Agents,  
14 South Square,  
Gray's Inn,  
London, W.C.1.

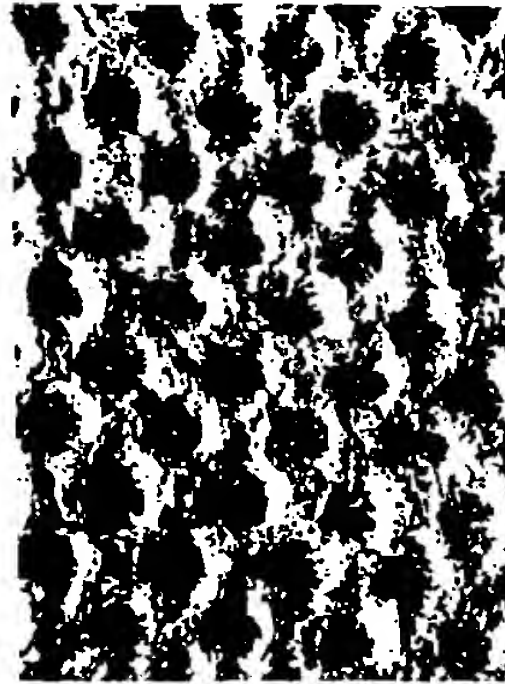
---

Printed for Her Majesty's Stationery Office by the Courier Press, Leamington Spa, 1970.  
Published by the Patent Office, 25 Southampton Buildings, London, WC2A 1AY, from  
which copies may be obtained.





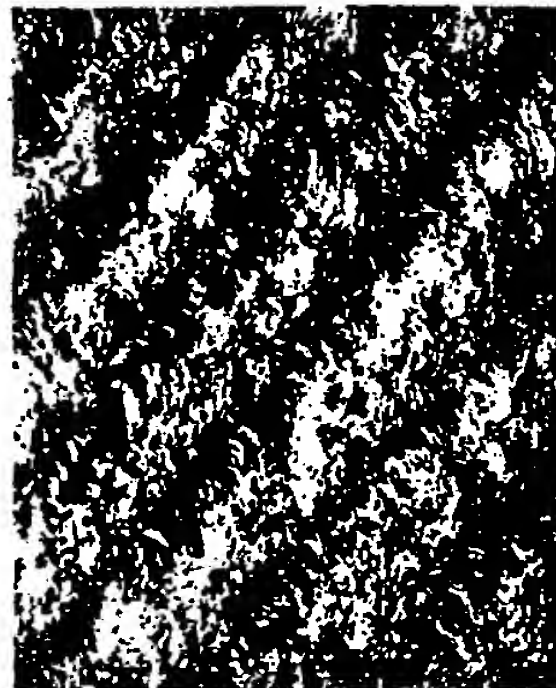
*FIG. 3*



*FIG. 4*



*FIG. 5*



*FIG. 6*

